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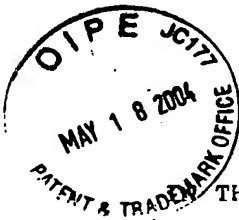
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THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Inventor: Kaoru ISOBE et al. ART Unit: 1751

Appln No.: 10/069,782

EXAMINER: Kopec, Mark T

Filed: February 28, 2002

For: ORGANIC SOLVENT-BASED DISPERSION OF CONDUCTIVE
POWDERS AND CONDUCTIVE PAINTDECLARATION UNDER 37 CFR 1.132

Sir:

I, Katsuichi CHIBA, do HEREBY DECLARE that:

I graduated from Applied Chemistry, Graduate School of Engineering, Osaka Prefecture University in March 1992 and entered Ishihara Sangyo Kaisha, Ltd. in April 1992;

I had been engaged in study and development of Functional Titanium Dioxide between April 1992 and February 2000;

I have been engaged in study and development of Electroconductive Material since March 2000;

I have an understanding and direct knowledge of the comparative test performed in accordance with the method described below. This test compared the dispersibility of the organic solvent-based dispersion and the transparency, conductivity and adhesion of the paint formed by the dispersion in accordance with the invention of the above-referenced patent application and a control dispersion and a control paint. Table 1 shows the results.

1. Object:

The object is to confirm that use of conductive tin oxide powders containing silicon oxide or tungsten oxide gives superior dispersibility as compared with use of conductive tin oxide powders containing neither silicon oxide nor tungsten oxide in an organic solvent-based dispersion of conductive powders comprising an organic solvent-based medium as a dispersion medium, conductive tin oxide powders coated with an organic metal coupling agent on the surface of tin oxide particles, and a dispersant, and a conductive paint obtained by blending said dispersion with a resin.

2. Place where experiment was conducted:

The laboratory of Technical Development Group of Inorganic Technique Control Division of Ishihara Sangyo K.K. (the laboratory of former Functional Material Group of Application Research Laboratory).

3. Experimental method:

1) Materials used for the experiment:

Organic solvent-based medium: Toluene of kanto extra pure grade reagent (manufactured by KANTO KAGAKU)

Organic metal coupling agent: Silane coupling agent (AZ-6177 manufactured by Nippon Unicar)

Conductive tin oxide powders:

(1) Spherical antimony-containing tin oxide containing silicon oxide.

(2) Spherical antimony-containing tin oxide containing tungsten oxide.

(3) Spherical antimony-containing tin oxide containing neither silicon oxide nor tungsten oxide.

Reagents:

Tin chloride ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ manufactured by Kishida Chemical Co., Ltd.)

Antimony chloride (SbCl_3 manufactured by Kishida chemical Co., Ltd.)

Sodium silicate (manufactured by Kishida Chemical Co., Ltd.)

Sodium tungstate ($\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ manufactured by Kishida Chemical Co., Ltd.)

Hydrochloric acid (35% aqueous solution manufactured by Goto Chemical Co., Ltd.)

Sodium hydroxide (25% aqueous solution manufactured by KANTO KAGAKU)

Dispersant: Cationic type dispersant (CB-50 manufactured by Toho Chemical Co., Ltd.)

Resin: UV curing acrylic resin (DPHA manufactured by Nippon Kayaku Co., Ltd.)

Polymerization initiator (DETX-S manufactured by Nippon Kayaku Co., Ltd.)

Polymerization accelerator (EPA manufactured by Nippon Kayaku Co., Ltd.)

2) Method of preparation of sample:

(1) A solution prepared by dissolving 234.5 g of tin chloride ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$) and 15.6 g of antimony chloride (SbCl_3) in 500 ml of a 5N hydrochloric acid and a 3N aqueous sodium hydroxide solution in which was dissolved 1.0 g/l of sodium silicate in terms of SiO_2 , were added in parallel to 5 liters of water at 90°C over a period of 30 minutes so that pH of the system was maintained at 6.0-7.0, thereby to produce a co-precipitate of tin oxide and antimony oxide. Then, hydrochloric acid was added to adjust the pH of the system to 3, and thereafter the co-precipitate was filtered off

and then washed until the resistivity of the filtrate reached 10000 Ω cm. The resulting cake was calcined at 600°C for 4 hours in an electric furnace and pulverized by a pulverizer to obtain spherical antimony-containing tin oxide powders containing silicon oxide and having a specific surface area of 78 m²/g. The content of silicon oxide was 2.5% by weight based on tin oxide.

(2) Spherical antimony-containing tin oxide powders containing tungsten oxide and having a specific surface area of 73 m²/g were obtained in the same manner as in the above (1), except for using a 3N aqueous sodium hydroxide solution in which was dissolved sodium tungstate (Na₂WO₄·2H₂O) in place of sodium silicate. The content of tungsten oxide was 2.5% by weight based on tin oxide.

(3) Spherical antimony-containing tin oxide powders containing neither silicon oxide nor tungsten oxide and having a specific surface area of 76 m²/g were obtained in the same manner as in the above (1), except that a 3N aqueous sodium hydroxide solution in which sodium silicate was not dissolved was used and the cake was calcined for 4 hours at 550°C in place of 600°C.

(4) In accordance with the procedure of EXAMPLE 1 of the present application, in a juice mixer was charged 100 g of spherical antimony-containing conductive tin oxide powders containing silicon oxide. While agitating, 12.5 g of 1/4 (in a weight ratio) solution of silane coupling agent AZ-6177 in ethanol was added to the powders followed by stirring. Then, the mixture was dried at 150°C for 40 minutes to coat the surface of the particles with the silane coupling agent. The amount of the silane coupling agent used for the coating was 2.5% by weight based on the spherical antimony-containing conductive tin oxide powders containing silicon compound. In a glass bottle, 40 g of the thus coated tin oxide powders, 3.2 g of cationic type dispersant CB-50, 70 g of toluene and 160 g of zircon beads were charged and ground for 90 minutes using a paint conditioner (Model #5110, made by Red Devil Co.) to give the organic solvent-based dispersion (Sample A). The solid content of Sample A was 38.2% by weight.

(5) The organic solvent-based dispersion (Sample B) was obtained in the same manner as in the above (4), except that spherical antimony-containing conductive tin oxide powders containing tungsten oxide and having a specific surface area of 73 m²/g were used in place of the spherical antimony-containing conductive tin oxide powders containing silicon oxide. The solid content of Sample B was 38.2% by weight.

(6) An organic solvent-based dispersion (Sample C) was obtained in the same manner as in the above (4), except that spherical antimony-containing conductive tin oxide

powders containing neither silicon oxide nor tungsten oxide and having a specific surface area of $76 \text{ m}^2/\text{g}$ were used in place of the spherical antimony-containing conductive tin oxide powders containing silicon oxide. The solid content of Sample C was 38.3% by weight.

3) Evaluation of the organic solvent-based dispersions:

(1) Evaluation on the dispersibility of the organic solvent-based dispersions:

The conductive tin oxide particles in the organic solvent-based dispersions (Samples A, B and C) were measured in terms of median size, using a particle size distribution measuring instrument, CAPA-700 (made by Shimadzu Seisakusho), which was used as an index of the dispersibility of the dispersions.

(2) Evaluation on dispersibility and conductivity of paint film made of conductive paint:

The solid contents of the organic solvent-based dispersions (Samples A, B and C) were diluted with toluene to 30% by weight. Seventeen grams of each of the diluted samples was mixed with 2.5 g of a UV curing acrylic resin (DPHA), a polymerization initiator (DETX-S), a polymerization accelerator (EPA) and 7.7 g of toluene, and the mixture was agitated to obtain a conductive paint.

The thus obtained conductive paint was coated on a glass plate in a dry thickness of $2.3 \mu\text{m}$ using a bar coater, subjected to natural drying and then exposed to UV rays to obtain a paint film. The surface resistivity and haze percentage (an index showing the degree of a cloudy appearance of a film; the lower the haze value, the higher the transparency) of the paint film were determined using a digital ohm meter (Model R-506, made by Kawaguchi Denki Seisakusho) and a haze meter (Model NDH-300A, made by Nippon Denshoku Kogyo K.K.), respectively. Furthermore, pencil hardness was evaluated according to JIS K5400.

The results are shown in TABLE 1. From the results of TABLE 1, it is recognized that in the organic solvent-based dispersion and the conductive paint obtained by blending the dispersion with a resin, use of conductive tin oxide powders containing silicon oxide or conductive tin oxide powders containing tungsten oxide gives dispersibility, transparency, conductivity and adhesion higher than those given by use of conductive tin oxide powders containing neither silicon oxide nor tungsten oxide.

Table 1: Dispersibility, transparency, conductivity and adhesion of organic solvent-based dispersion and conductive paint

Samples	Dispersed state of dispersion	Median size of dispersion (μm)	Surface resistivity (Ω/\square)	Haze percentage (%)	Pencil hardness
Sample A	Good	0.10	1.5×10^5	0.62	4H
Sample B	good	0.12	2.0×10^5	0.52	4H
Sample C	good	0.16	1.0×10^{10}	2.22	2H

4. Conclusion

From the above results of evaluation, it is confirmed that in an organic solvent-based dispersion of conductive powders comprising an organic solvent-based medium as a dispersion medium, conductive tin oxide powders coated with an organic metal coupling agent on the surface of tin oxide particles, and a dispersant, and a conductive paint obtained by blending said dispersion with a resin, use of conductive tin oxide powders containing silicon oxide or tungsten oxide gives dispersibility superior to that given by use of conductive tin oxide powders containing neither silicon oxide nor tungsten oxide.

The undersigned declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 11th day of May, 2004.

Katsuichi Chiba
Katsuichi CHIBA